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HCl Etching Behavior on Low-Tilt-Angle and Step-Free 4H-SiC Surfaces

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Abstract. We report on new observations made, when 4H-SiC, Si-face substrate mesas, having either low tilt-angle (< 1°) with steps or step-free top surfaces, were exposed to three separate HCl etching conditions for five minutes at temperatures of 1130°C, 1240°C and 1390°C. We observed that HCl was ineffective at 1130°C, as etching was incomplete with abundant surface contamination. At 1240°C, screw dislocations were aggressively etched by HCl, while multiple shallow flat-bottomed etch pits were formed on step-free mesa surfaces. At 1390°C, step-flow etching dominated as large etch pits were formed at screw dislocations and previously step-free surfaces etched inward from mesa edges to form parallel rows of organized steps.

Introduction

In order to obtain high surface step density to ensure correct epitaxial polytype replication during epitaxial growth, most commercial 4H-SiC wafers are typically cut with an off-axis tilt angle of 4° to 8°. This offcut angle results in a significant amount of boule waste that increases with increasing wafer diameter. There is increasing interest in homoepitaxial growth on low-tilt-angle (i.e., less than 1° and commonly called on-axis) 4H-SiC substrates. It is well known that proper preparation of on-axis substrates prior to the initiation of epitaxial growth is required to prevent nucleation of 3C-SiC [1-3]. HCl has been used as an effective pregrowth etchant to remove latent crystal damage from the substrate surface prior to epitaxial growth initiation [1-5].

Low-tilt angle surfaces that are produced by epitaxial growth hillocks at screw dislocations (SDs) can approximate the step structure of commercially available on-axis wafers and are comprised of step edges and step terraces [1]. In contrast, the topmost surface of the step-free mesa surface is a large-area (0001) basal plane surface devoid of atomic-scale steps [1, 6]. These two types of mesa surfaces (i.e., mesas with SD assisted growth hillock steps and step-free mesas) reside arrayed across a single wafer and are thus subjected to identical etching process conditions. This paper reports on new observations made on the effects of HCl etching on low-tilt angle mesas (with SD growth hillocks) and step-free surfaces with respect to temperature.

Experimental

In order to produce substrates that contain a mix of low-tilt angle and step-free mesa surfaces, a commercially available on-axis 4H-SiC Si-face wafer was patterned with an array of small rectangular (e.g., 50 μm x 200 μm x 5 μm deep) mesas by dry etching techniques. The wafer was diced into quarters. One of the quarter wafers was subsequently subjected to the step-free mesa growth process more fully described in [1, 6]. A pregrowth in-situ hydrogen etch of 5 minutes at 1530°C was followed immediately by twenty minutes of pure step-flow growth conditions at 1535°C in a commercial horizontal flow cold wall chemical vapor deposition (CVD) system using an inductively heated TaC coated graphite susceptor. The growth phase used silane (SiH_4) and propane (C_3H_8) with a Si/C = 1 in a 8000 l/min hydrogen carrier gas. After growth, the substrate was characterized by Atomic Force Microscopy (AFM) to confirm that the top surfaces of mesas not threaded by a substrate SD were free of SiC bilayer steps. For mesas threaded by a SD, a growth hillock forms at the SD such that the top surface of that mesa has steps and a low-tilt angle. The substrate was diced into 3mm x 4mm die for subsequent HCl etching experiments.

Three consecutive HCl etching runs listed in Table 1 were performed. All three runs were terminated in exactly the same manner, a cool down rate from etching temperature to the 600°C detection limit of the optical pyrometer of ~5.2°C/sec (with further cooling to room ambient) with HCl and H_2 present then switching to a 100% argon purge. AFM analysis was conducted on the tops of mesa surfaces, for all three conditions.

Table 1. HCl etching runs

RUN	Temp. (°C) ± 10°C	Time (min)	Pressure (millibar)	HCl (sccm)	H_2 (sccm)
1	1130	5	200	50	4000
2	1240	5	200	50	4000
3	1390	5	200	50	4000

Results

At 1130°C, no organized etching behavior was observed on either low-tilt-angle (SD-stepped mesa surfaces) or step-free mesa surfaces. Particulates were observed on both step-free and SD-stepped surfaces. However, the particulate density was extremely high for SD-stepped mesa surfaces, as shown in Fig 1a. Auger analysis suggests the particles may be carbon but the results were inconclusive. The source of the particles has not yet been isolated. On step-free surfaces (Fig 1b), particles were still present but were more than 100X lower in density. HCl etching does not appear to be effective at 1130°C, leaving a contaminated surface and no evidence of etching of the SiC surface.

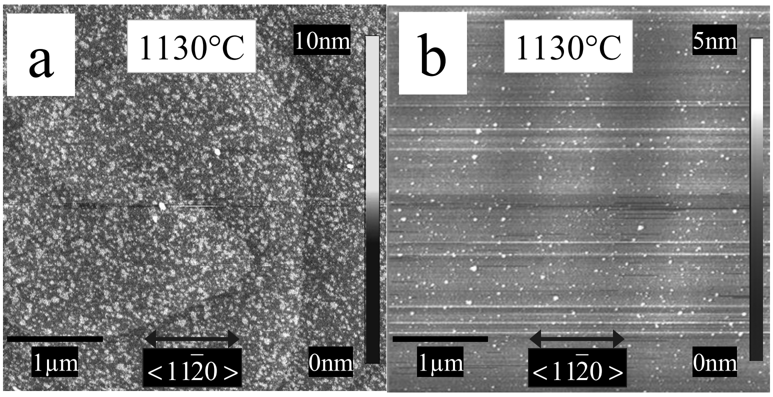

Fig 1 a) AFM of SD core etched at 1130 °C, resulted in a surface with very high surface contamination. b) AFM scan of step-free surface with particles present. Horizontal lines are artifacts of the AFM. There is no evidence of effective HCl etching on either surface.

At 1240°C two distinct etching behaviors were observed, depending upon the mesa step structure/defect content. The enhanced etching at the core of SDs resulted in aggressive downward etching, leading to a pointed-bottom etch pit formation, Fig 2a,b. Once enhanced (dislocation-assisted), downward etching of a bilayer creates a new bilayer step edge, continued etching of that step edge then proceeds in a lateral (step-flow) manner. The high pass filtered AFM image of figure 2a illustrates the smooth step edges exposed during dislocation assisted etching while remnants of growth hillock step edges are ragged. The resulting SD etch pits were >100nm deep and ~3-4μm in diameter at the surface of the substrate; the angle of the etch pit sidewall is ~5°, Fig 2b. It was not possible to determine the exact depth of the etch pit, as the AFM probe tip could not scan the bottom of the etch pit.

Unlike the defect-assisted etching that was observed at SDs, the 1240°C etching of previously step-free mesa surfaces resulted in the formation of multiple shallow flat- bottomed pits, Fig 2c,d. Prior to etching, the topmost surface of a step-free mesa is a (0001) basal plane surface. In the absence of a dislocation to assist downward (c-axis) etching through crystal bilayers, the downward etching progresses at a much reduced rate as evidenced by the shallow depth (~10nm) and low sidewall angle (~0.16°) of the etch pits, Fig 2d. However once step-edges are created by downward etching through crystal bilayers, this etching of existing step-edges results in a flat-bottomed pit, Fig 2c,d. Note that the particulate contamination that plagued the surfaces at the lower etch temperature of 1130°C is almost nonexistent at 1240°C. The locations of etch pit formation on mesa surfaces appears to be random. However, it was not determined what mechanism was the driving force for local etch pit development.

At 1390°C, step flow etching produces well ordered step trains on both low-tilt-angle and step-free mesa surfaces. Figure 3a depicts results when a single SD resides within a mesa (Fig 3a inset photo). A large diameter (~30-40µm) etch pit forms at the SD, Fig 3b. Note that the angle of the pits sidewall facet is ~0.8° (Fig 3b) much smaller than the angle formed at a similar SD by 1240°C etching, Fig 2a,b.

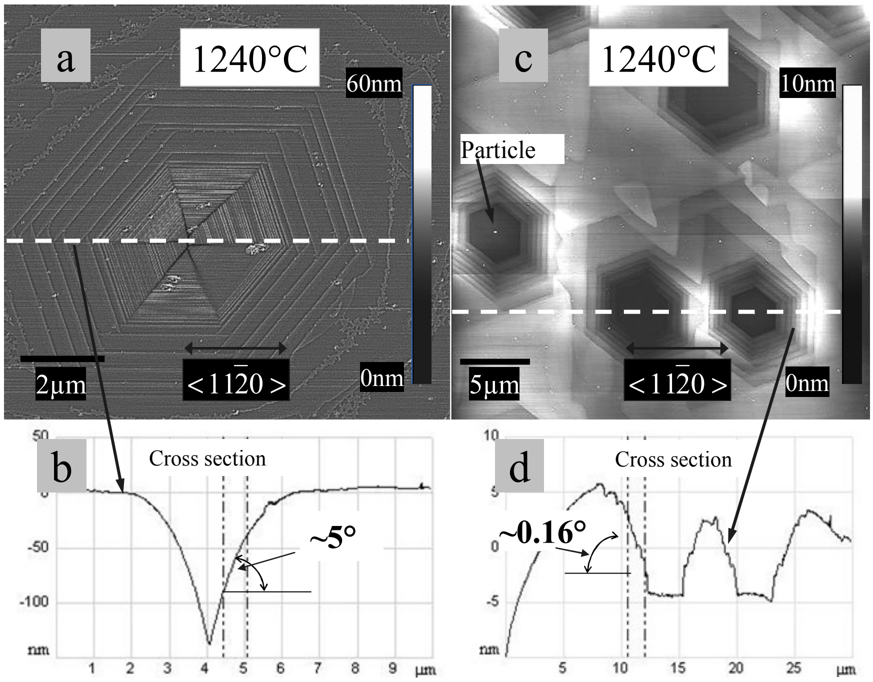

Fig 2 a) High pass filtered AFM image of the etched SD showing cross section path and step structure. b) AFM cross section analysis of SD etched at 1240°C, a deep narrow etch pit forms at the core of the SD. Note that the angle of the sidewall is ~5°. c) AFM image of the etched mesa showing path of cross section and multiple pits d) AFM cross section analysis of a previously step-free mesa etched at 1240°C. Multiple shallow flat bottomed etch pits have formed. Note that the angle of the sidewall for the shallow pits is only ~0.16° also note that change in scales of the y-axis.

Previously step-free surfaces exposed to 1390°C etching conditions resulted in the formation of roughly parallel steps etching inward from the mesa edges, Fig 3c,d. A roof top like morphology evolves as shown in the inset photo of Fig 3c. No etch pits were observed to form on any of the step-free surfaces under these conditions. The angle formed by the well ordered step trains is ~0.5°, Fig 3d. On both low-tilt angle surface and step free mesas the surface appears to be free of particulates.

It is important to note that, the step-to-step distances and local tilt angles found for both 1390°C step trains are quite similar. This occurs despite the fact that the mechanism responsible for introduction of new step edges to these trains is quite different. Enhanced downward etching of bilayers at the dislocation provides step edges for the pit, while non-uniform attack of the mesa sidewall supplies step edges moving inward for dislocation free mesas. The similarity of both surfaces suggests the surface structure in these etching conditions may be governed by a mechanism other than rate of step creation by downward etching through bilayers.

The above results are additional evidence that step-to-step repulsive interaction is a key driving mechanism behind well-ordered step trains. We previously proposed this variation of Ohtani's model [7] to explain hydrogen etching of step-free mesas [8]. We propose here that the same repulsive interaction may be dominating the step structure of the SD etch pits formed by HCl etching at 1390°C. This interaction mechanism is clearly not dominant at 1240°C.

Conclusion

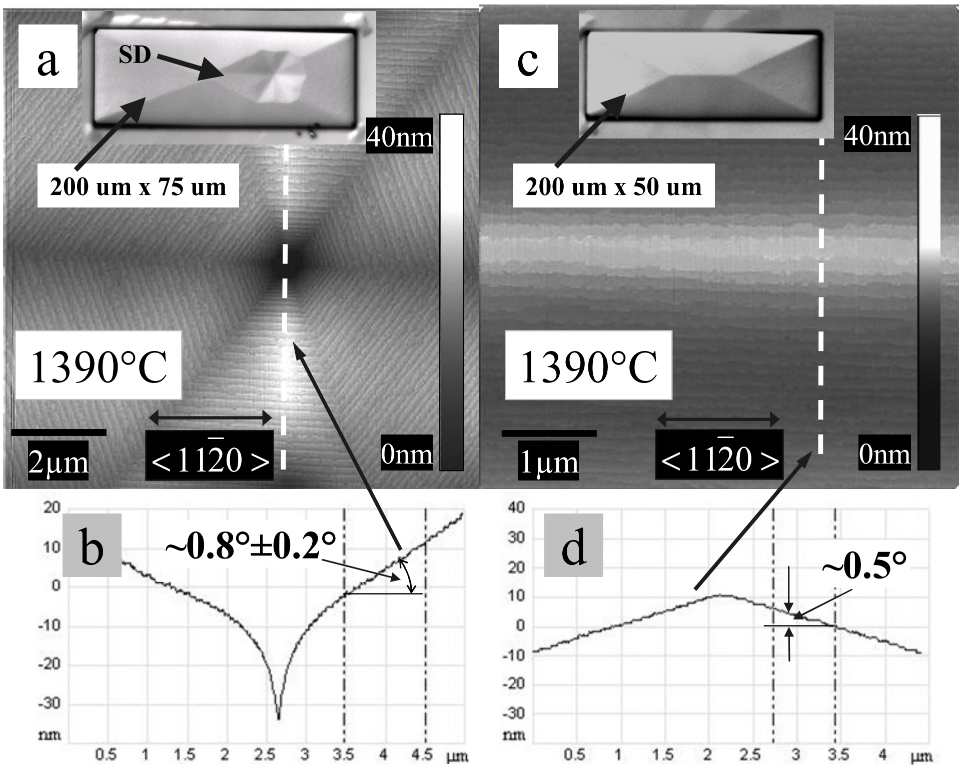

Fig 3 a) AFM image of the etched SD showing path of cross section, inset is optical photo of mesa. b) AFM cross section analysis of SD etched at 1390°C, a deep wide etch pit forms at the core of the SD. Note that the angle of the sidewall is ~0.8°. c) AFM image of the etched mesa showing path of cross section with parallel steps, inset is optical photo of mesa with roof top morphology. d) AFM cross section analysis of a previously step-free mesa etched at 1390°C. Note that the well ordered step trains form an angle of ~0.5°.

HCl can be an effective etchant for on-axis SiC substrates. However, we have observed that at lower temperatures (1130°C) neither low-tilt angle nor step-free surfaces are etched effectively and the resultant surfaces are contaminated with particulates. At an intermediate temperature (1240°C) dislocations are effectively etched, but step-free (i.e., basal plane surfaces) are attacked resulting in the formation of multiple shallow etch pits. At the higher temperature of 1390°C downward etching of dislocations and step free mesas result in similar step densities indicating that step-to-step repulsive forces become dominant at these higher temperatures. Particulate removal is effective and complete. This work extends previous observations of HCl etching on 6H on-axis substrates at atmospheric pressure [1, 2, 4, 6].

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